

**Albuquerque Bernalillo County****Water Utility Department**WATER RECLAMATION DIVISION  
4201 2ND STREET SW, ALBUQUERQUE, NEW MEXICO 87105**WATER QUALITY LABORATORY  
STANDARD OPERATING PROCEDURE APPROVAL FORM**WQL SOP **225 Total Dissolved Solids**CURRENT REVISION # **04** DATE **November 7, 2007**ORIGINAL ISSUE DATE **February 1998****MODIFICATIONS AND REASONS FOR REVISION**

*Changes to procedure – See Sections 6.1 and 6.2
*Section 6.2 – Replace use of aluminum evaporating dish with glass or porcelain
* Section 6.2 – Changed drying time from 24 hours to 1 hour
* Changes mandated due to continuous “Not Acceptable” results for WP/WS samples
* Verified procedures with 19th ed. Standard Methods 2540 C
* Laboratory Analysts training on file in QA File Rm

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Out of Service By: \_\_\_\_\_ Date: \_\_\_\_/\_\_\_\_/\_\_\_\_

Reason: \_\_\_\_\_

## STANDARD OPERATING PROCEDURE

### SOP 225

### **TOTAL DISSOLVED SOLIDS**

**SCOPE AND APPLICATION:** This method is applicable to drinking, surface and saline waters, domestic and industrial wastes. The practical range of determination is 10 mg/L to 20,000 mg/L.

#### APPLICABLE METHOD REFERENCES:

EPA 1984, storet number 70300, residue, total filterable at 180°C  
Std.Methods 19th ed., 2540C

**DISPOSAL OF SAMPLES:** Dispose of samples at the sample compositing sink, located in sample preparation area. This sink bypasses the acid trap and goes directly to the sanitary sewer.

## TOTAL DISSOLVED SOLIDS

### 1.0 GENERAL DISCUSSION

**1.1 Principle:** A well-mixed sample is filtered through a standard prepared glass-fiber filter and the filtrate is evaporated and dried to constant weight at 180°C.

**1.2 Interferences:** Highly mineralized water containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200mg.

**1.3 Safety Considerations:** Samples may be bio-hazardous avoid ingestion, inhalation and contact with the skin. Sample dishes are heated to extreme temperatures, avoid contact with the skin or severe burns may result. Use heat resistant gloves, tongs and eye protection when placing or removing sample dishes from oven.

**1.4 Sample preservation & Storage:** Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to container walls. Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours. In no case hold sample more than 7 days.

**1.5 Sample Preparation:** Bring samples to room temperature before analysis.

**1.6 Method Performance Criteria:** The reference method cites single-laboratory analyses of 77 samples of a known sample, 293 mg/L were made with a standard deviation of differences of 21.2 mg/L.

**2.0 Apparatus & Equipment:** Drying oven, Desiccator (<10 RH), analytical balance (mettler AE-100), wide-mouth pipets, vacuum support manifold, magnetic filtering funnels, magnetic stirrer and magnetic stirring bar, forceps.

**3.0 Reagents & Supplies:** Glass fiber filters (Whatman 934-AH 47mm), disposable aluminum dishes 9mm X 50mm, deionized water, glass or porcelain evaporating dish 100-140ml capacity. **NOTE:** Aluminum drying pans are no longer acceptable for use following this SOP.

#### **4.0 Quality Control Procedure:**

**4.1** The balance must be checked with type II weights weekly and the results logged in the balance log. Annually the balance must be serviced and calibrated by a certified service person. If the type II weight check is out of the acceptable range (acceptability criteria in balance log) or the balance annual service is not conducted all results subsequent to the last satisfactory type II weight check or one year expiration date of the last annual service of the balance, will be qualified in LIMS by text on the samples.

**4.2 Laboratory Reagent Blank (LRB)** will be conducted on each batch of samples. The control blank should have a quantification of zero mg/L TDS. If the quantification of the control blank is greater than 2 mg/L TDS, then all samples in the batch are to have the results qualified when reported. Qualify the results by text on the samples and stating that the control blank was greater than 2 mg/L. Results also must be qualified by text on the samples in LIMS, if the drying oven is out of range ( $180^{\circ} \pm 2^{\circ}\text{C}$ ), or analytical balance won't calibrate.

**4.3 Laboratory Control Sample (LCS)** is an aliquot of reagent water or other blank matrices to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The laboratory must analyze at least one LCS with each batch of samples. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judged out of control, and the source of the problem should be identified and resolved, any corrective action taken must be documented in the bound work sheet book concomitant with the analysis data. The laboratory must use LCS analyses data to assess laboratory performance against the required control limits of 85-115%. When sufficient internal performance data become available, usually a minimum of 20-30 analyses, optional control limits can be developed from the percent mean recovery ( $\bar{X}$ ) and the standard deviation ( $S$ ) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = \bar{X} + 3S$$

$$\text{LOWER CONTROL LIMIT} = \bar{X} - 3S$$

three successive 20-mL portions of reagent-water. Continue suction to remove filter from filtration apparatus and transfer to an aluminum dish. Take care to prevent the dried filter from adhering to the 9mm X 50mm aluminum dish. Store in desiccator until needed.

**6.2 Preparation of evaporating dish:** Heat clean glass or porcelain evaporating dish to  $180^{\circ}\pm 2^{\circ}\text{C}$  for 1 hour in an oven. Store in desiccator until needed. Weigh immediately before use. **NOTE:** Aluminum drying pans are no longer acceptable for use following this SOP.

**6.3 Selection of filter and sample sizes:** Choose sample volume to yield between 10 and 200 mg dried residue. Initially, use a 100mL sample volume for most sample unless otherwise contraindicated. If more than 10 minutes are required to complete filtration, increase filter size or decrease sample volume. When very low total suspended solids are encountered (less than 10 mg/L), less dried residue may be collected; compensate by using a high-sensitivity balance (0.002 mg).

**6.4 Sample analysis:** Stir sample with a magnetic stirrer or by hand, and while stirring, pipet a measured volume (usually 100mL) onto the seated glass-filter with applied vacuum. Wash with three successive 10-mL volumes of reagent-grade water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete. Samples with high dissolved solids may require additional washings. Transfer total filtrate with washings to a weighed evaporating dish and evaporate to dryness in a drying oven. If necessary, add successive portions to the same dish after evaporation. Dry evaporated sample initially for a least 1 hour in an oven at  $180^{\circ}\pm 2^{\circ}\text{C}$ , cool in a desiccator to balance temperature, and weigh. Repeat drying cycle (1 hour minimum after initial drying cycle) of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4% of previous weight or 0.5 mg, whichever is less.

**7.0 Calculation:**

$$\text{mg total dissolved solids/L} = \frac{(A - B) \times 1000}{\text{sample volume mL}}$$

where:

A = weight of dish + dried residue, mg.

B = weight of dish, mg.

**8.0 Reporting:** All measurements and results will be recorded in the bound work sheet book for Total Dissolved Solids. The determined results for each sample tested will be entered on the electronic data system, SQLIMS. All samples requiring qualification will be text at the sample level in SQLIMS. All analyses requiring corrective actions will have the documentation of the corrective action in the bound work sheet book, concomitant with the sample results, and QC result.

The optional control limits must be equal to or better than the required control limits of 85-115%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LCS. These data must be kept on file and be available for review. If the LCS is not within its control limits, then the affected samples must have text to qualify the data. The text should state the percent recovery achieved for the LCS. Preparation of a LCS 250mg/L TDS: Weigh out 0.25g NaCl (ACS grade) dissolve in 200 mL reagent water and dilute to 1000 mL.

**Calculation of percent recovery for an LCS:**

$$R = \frac{LFB - LCS}{s} \times 100$$

where: R = Percent recovery

LRB = Laboratory reagent blank.

LCS = Laboratory control sample

s = Concentration equivalent of analyte added to fortify the LRB solution.

**4.4 Laboratory Control Sample Duplicate (LCSD)** is prepped exactly like the LCS and measures precision of the methodology.

**4.5 Control Charts-** All quality control data will be entered in the lab share drive by analysts performing this test. Quality assurance reviews are performed weekly, for complete details of control chart performance evaluations see QA SOP-005.

**5.0 Instrument Calibration:** Mettler AE-100 Analytical Balance. Press and hold the single control bar until -CAL- appears in the display, then release control bar. The display changes to CAL---, then to CAL 100 (blinks). Move calibration lever all the way to the rear; the display changes to CAL---, followed by 100.0000. Move calibration lever all the way back towards the front of the balance; the display changes to ---, followed by 0.0000. If CAL Err appears in the display, the weighing pan was not unloaded before calibrating the balance, or the wrong external calibration weight was used (return to the weighing mode by pressing and holding the control bar). If No CAL appears in the display, a temporary malfunction has occurred (recalibrate balance). Calibration of the instrument is to be conducted prior to use.

**6.0 Procedure:**

**6.1 Preparation of glass-fiber filter:** Insert filter with wrinkled side up in filtration apparatus. Apply vacuum and wash filter with